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CAPACITOR DISCHARGE THERMAL INITIATION
APPARATUS FOR HIGH ENERGY MATERIALS

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CAPACITOR DISCHARGE THERMAL INITIATION APPARATUS
FOR HIGH ENERGY MATERIALS

Prepared by:
Paul Kendall and
Jerome Rosen

ABSTRACT: A thermal initiation apparatus is fully described for rapidly heating and monitoring high energy materials in the range of 300 to 1000°C. Time delays to explosion, which range from 0.05 to 5 msec are a function of sample temperature. A capacitor discharge pulse heats the sample enclosed in a stainless steel hypodermic needle tubing. A constant current source is used to monitor the tubing resistance and therefore to determine its temperature. Calibration methods, sample handling techniques, and representative thermal initiation data are also reported.

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U. S. NAVAL ORDNANCE LABORATORY
WHITE OAK, MARYLAND

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19 June 1968

**Capacitor Discharge Thermal Initiation Apparatus for
High Energy Materials**

Studies at the Naval Ordnance Laboratory have been carried out to gain further information on the sensitivity of organic high explosive and propellant materials. A thermal sensitivity method has resulted in which small, highly confined samples are heated very rapidly to temperatures in the range of 300 to 1000°C. This report describes the design features and operation of the capacitor discharge thermal sensitivity apparatus in use at the present time. This work was performed under ORD 033 221 F008 08 11 Prob 005, Desensitization of Explosives.

E. F. SCHREITER
Captain, USN
Commander

Albert Lightbody
ALBERT LIGHTBODY
By direction

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REFERENCES

- (a) Wenograd, J., Trans Faraday Soc, 57, 1612 (1961)
- (b) Rosen, J. M., Holden, J. R., Div of Fuel Chem, ACS, 7, 35 (1963)
- (c) Kendall, P. A., "Longitudinal Vibration Induced in Metals by Thermal Shock", Applied Physics Letters, to be published May 1968

INTRODUCTION

Studies at the Naval Ordnance Laboratory have been carried out to gain further information on the sensitivity of organic high explosive and propellant materials. A thermal sensitivity method has resulted in which small, highly confined samples are heated very rapidly to temperatures in the range of 300 to 1000°C (references (a), and (b)). The time delays to explosion, which fall in the range of 50 microseconds to a few milliseconds, are measured as a function of the sample temperature. This report describes the design features and operation of the thermal sensitivity apparatus in use at the present time.

In the thermal sensitivity measurement, a 2.1 microliter sample is enclosed in a 6.35 cm length of stainless steel hypodermic needle tubing. The tubing is then heated very rapidly by discharging a capacitor through it, and its resistance is measured as a function of time. The temperature to which the tubing has been raised by the capacitor discharge is determined from the ratio of its hot resistance to its room temperature resistance. When the sample within the tube explodes, it bursts the tube wall causing an abrupt change in the resistance of the tube. Therefore, both the temperature of the sample container and the delay time before explosion are determined by measuring the resistance of the hypodermic needle tubing as a function of time. This is done by passing a constant current through the tube and monitoring the voltage across it with an oscilloscope (CRO). The delay time also is measured with an electronic timer which is started by a signal from the capacitor discharge and stopped by a signal from a microphone located near the bursting sample tube.

GENERAL CIRCUIT DESCRIPTION AND DESIGN FEATURES

The thermal initiation apparatus is shown in Figure 1 in semi-block diagram form. It consists of a series high current discharge circuit for pulse heating the explosive sample, R_0 , and auxiliary circuitry and equipment to perform the required measurements and monitoring. The programmer is manually started. It then automatically controls the thermal initiation apparatus from the charging of C_0 to the exhausting of the gases from the exploded sample. In the 20 millisecond period before capacitor discharge the CRO sweep, calibrator, and constant current supply are activated sequentially in that order. The CRO horizontal sweep is triggered producing a zero voltage reference trace. The calibrator which is preset to equal the expected signal across the explosive sample switches to the -B input. The constant current unit supplies power to R_0 about one millisecond before capacitor discharge and remains in that state for about 20 milliseconds. At capacitor discharge the CRO sweep is again triggered. The signal across the sample minus the calibrator voltage (A-B) provide essentially null operation. This makes it possible

to operate the CRO at a high vertical sensitivity, usually about 20 mv/cm. The resistance at ambient temperature, R_0 , is measured prior to automatic operation. This is done by using a very low steady state constant current and monitoring the voltage across R_0 with a L&N potentiometer. The condition of low current and the 20 millisecond powering time during automatic operation is required in each case to avoid resistance change problems due to heating.

The component values listed for the discharge circuit shown in Figure 1 were chosen for a slightly underdamped condition. This insured a positive cutoff for the 5C22 and, therefore, a clean negative current pulse through R_0 . A maximum capacitor voltage of about 3000 volts (≈ 1600 ampere discharge) is required to increase R_0 from 0.6Ω at room temperature to 1Ω which corresponds to about 1000°C . The discharge period lasts about 20 μsec . The constant current supply consists of a series resistance, a fast acting mercury relay and a solid state 54 volt power supply. The series resistance consists of two 25Ω parallel resistors for automatic operation and switches to a 1000Ω component for initial R_0 measurement. The solid state power supply is protected from the high voltage discharge pulse by a 16,000 μF capacitor bank. The capacitor bank, non-inductive resistors, and a fast acting mercury relay are located very close to R_0 . This is done to minimize the inductance of the interconnecting wires since the inductance of the constant current unit adversely affects the recovery time of the system from the high voltage pulse. The CRO is satisfactorily isolated from the high negative voltage pulse by the simple diode clipping circuit shown in Figure 1. It clips the high negative voltage pulse and passes, essentially unattenuated, the positive dc signal. This clipping diode has a relatively slow response but was chosen because it has no overshoot. However, this diode is adequate for studies down to 50 μsec . In addition for reliable operation all connections were soldered in the high current discharge circuit with the exception of the two screw clamps used to hold the sample explosive. This was particularly important for those connections exposed to the corrosive gases of the exploding samples. The stainless steel tube holding the explosive sample was soldered to two small brass posts that in turn were securely held by the two screw clamps.

An exploding wire record is shown in Figure 2 from which the delay time to explosion, and indirectly temperature measurements are obtained. It should also be noted that the trace oscillation seen in the figure is a mechanical longitudinal motion of the stainless steel sample tube (reference (c)). We assume that the oscillations result from the rapid bulk expansion of the metal due to heating and because of its elastic nature reacts as a mechanical oscillator.

CALIBRATION

A resistivity-temperature* curve of the hypodermic needle tubing was determined by measuring the resistance at several different

*Resistivity is here defined as the ratio of the hot resistance, R_1 , to the room temperature resistance, R_0 .

temperatures, Figure 3. Fifty centimeter sections of the tubing were heated in a well insulated muffle furnace. At equilibrium, the resistances were measured along with that of a platinum resistance thermometer. Above 754°C we were not able to achieve equilibrium conditions probably because of both oxidation and slow annealing of the tubing. Each point represents the average from measurements made on three sections of tubing. The extrapolated part of our curve was in good agreement with previous data obtained with an optical pyrometer (reference (a)).

Several calibration resistors were made from #20 manganin wire to cover, adequately, the complete range of resistance found for heated sample tubes--about 0.6 to 1.0 ohm. Their resistance values were measured accurately on a Kelvin bridge and rechecked from time to time. The null voltage of each resistor was measured in the circuit in the automatic mode with the exception of capacitor discharge to obtain a voltage-resistance calibration curve. This relationship was then used to determine the resistance of the heated sample tube, R_1 , from the monitored voltage on the oscillogram.

For convenience, temperature is computed by an IBM 7090 digital computer using a program written for that purpose. However, it may be obtained from the temperature-resistivity calibration after first obtaining the resistivity from measured values of R_0 and R_1 .

SAMPLE HANDLING

The sample tubes are filled with a liquid by placing the open ends in the liquid as shown, Figure 4. The other ends are closed by spot welding. Air is removed from the tubing by a vacuum pump. When the atmosphere is readmitted, the liquid is forced into the tubing.

Essentially the same system is used for solids except that solids must be heated above the melting point. A silicone liquid bath is used. Thermal sensitivity measurements cannot be made on materials which decompose rapidly in the liquid state.

REPRESENTATIVE DATA

Typical data plots of the logarithm of the delay time to explosion as a function of the reciprocal of the absolute temperature are shown in Figure 5 for three representative liquids. In addition to computing temperatures, the program yields a least square fit of the data to the straight line defined by

$$\log_{10} \text{ delay time in milliseconds} = A + \frac{1000 B}{T, ^\circ K}.$$

One way to compare thermal sensitivities is to examine the temperatures required for initiation at a given time delay. These are listed in Table I for a heating period of 250 microseconds, a convenient point of reference. If a thermal pulse of 422°C is

generated for 250 microseconds we should expect triethylene glycol dinitrate to be initiated. These same conditions of time and temperature would not have a significant effect on nitromethane and n-propyl nitrate. As triethylene glycol dinitrate is the easiest to initiate, we conclude it is the most sensitive under the conditions described.

Sensitivity ranking by the Wenograd thermal initiation method are generally in good agreement with ranking by other sensitivity methods generating thermal pulses in the range of 0.1 to 1 millisecond.

ACKNOWLEDGMENT

The computer program was written by J. R. Holden and C. Dickinson. We are grateful for the assistance of Eleanore Kayser and Patrick Barry.

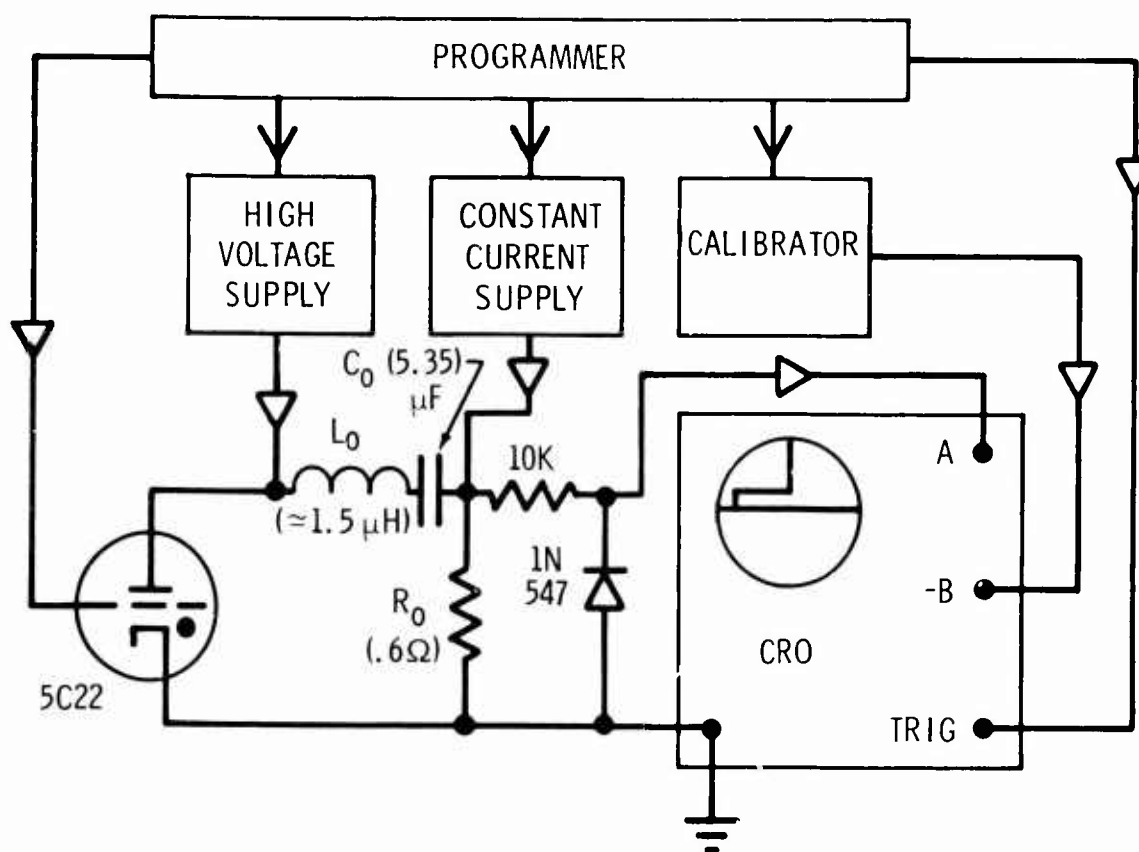
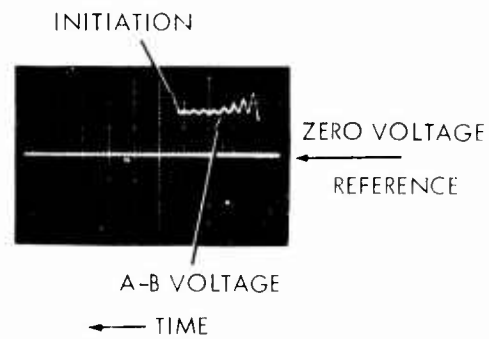


FIG. 1 THERMAL INITIATION APPARATUS



SWEEP SPEED = $50 \mu\text{SEC}/\text{CM}$
VERTICAL SENSITIVITY = $20 \text{ MV}/\text{CM}$
CAPACITOR DISCHARGE = 2230 V

FIG. 2 OSCILLOGRAM RECORD OF INITIATION

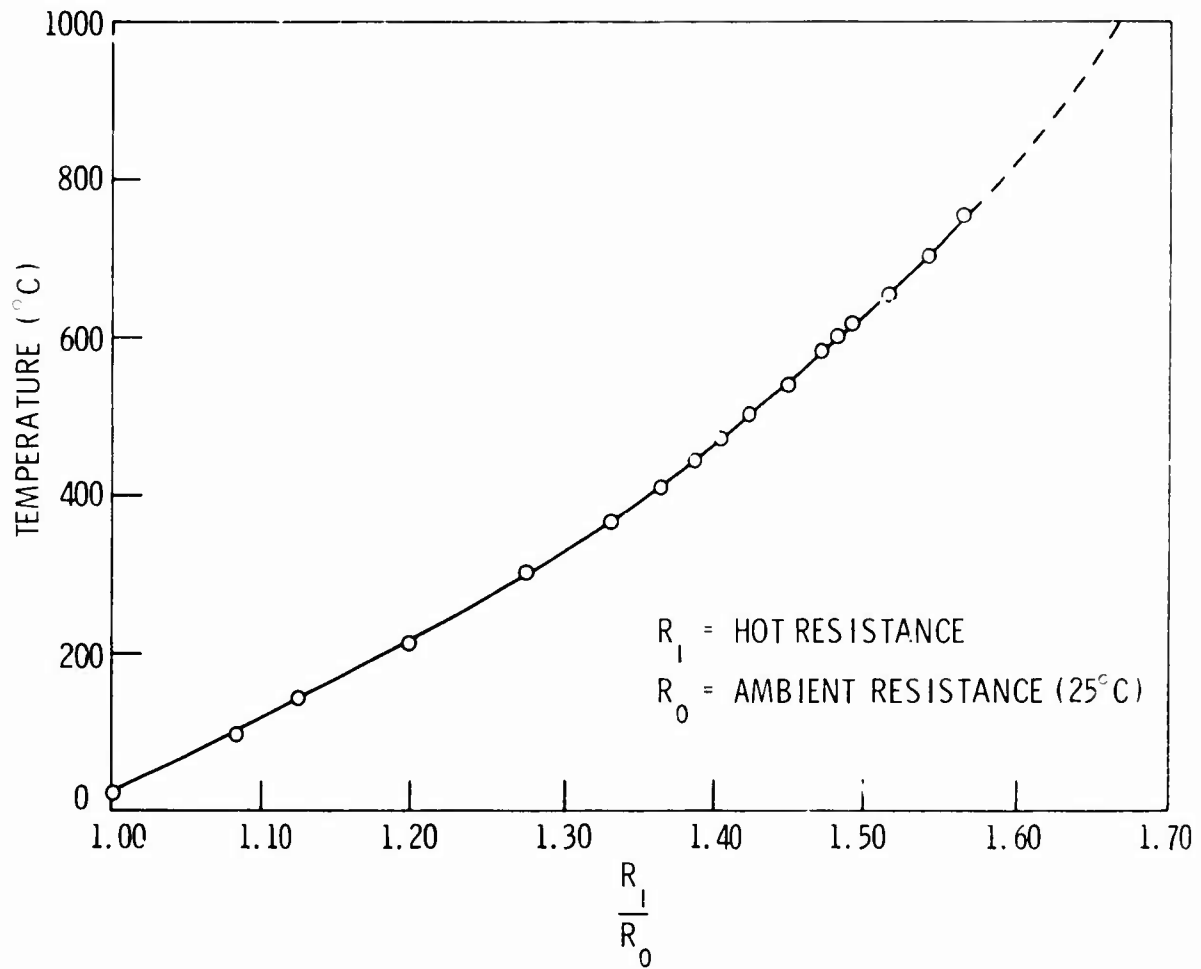


FIG. 3 TEMPERATURE-RESISTIVITY CALIBRATION
 #28 STAINLESS STEEL HYPODERMIC NEEDLE TUBING

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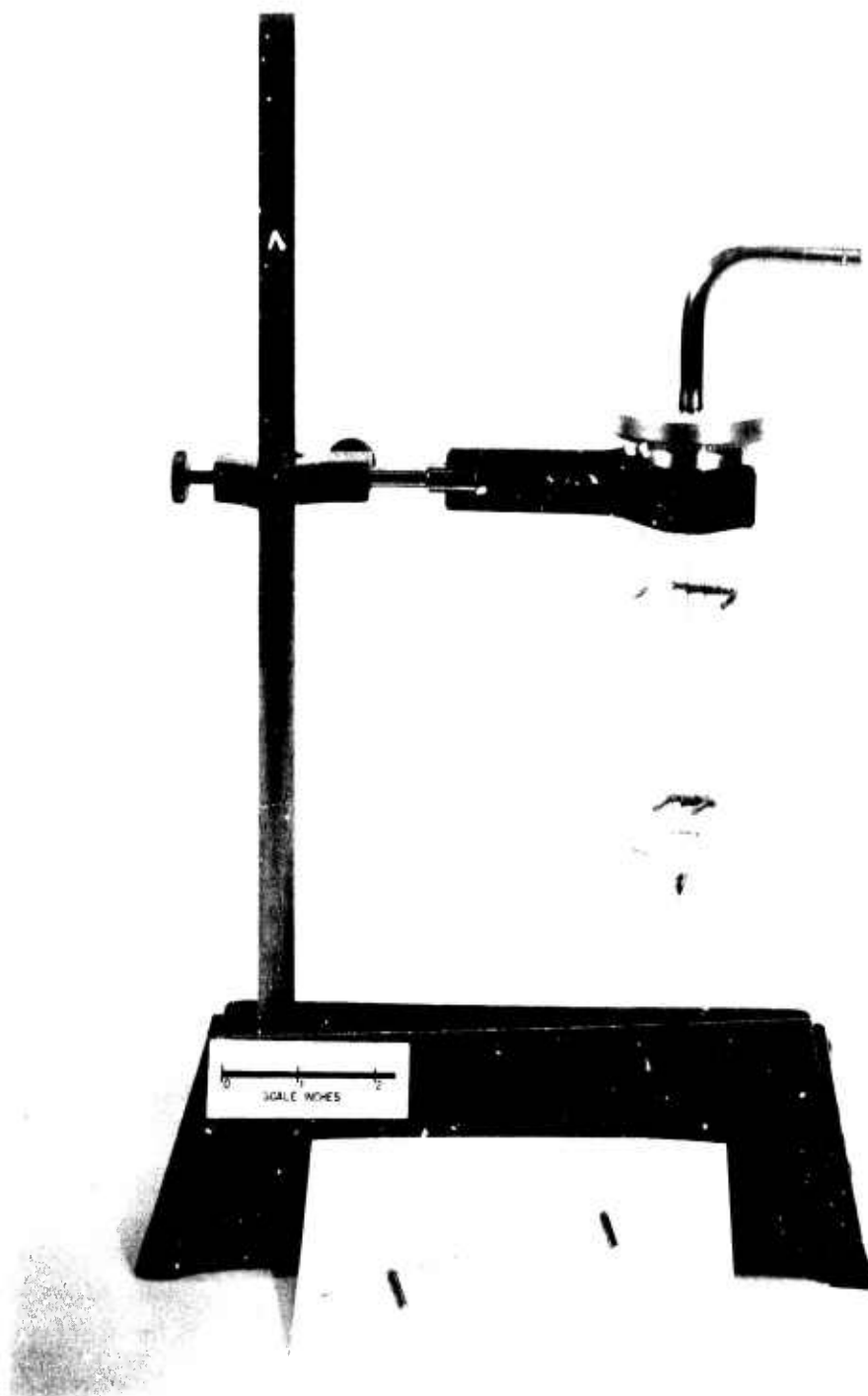


FIG. 4 SAMPLE LOADING TUBE

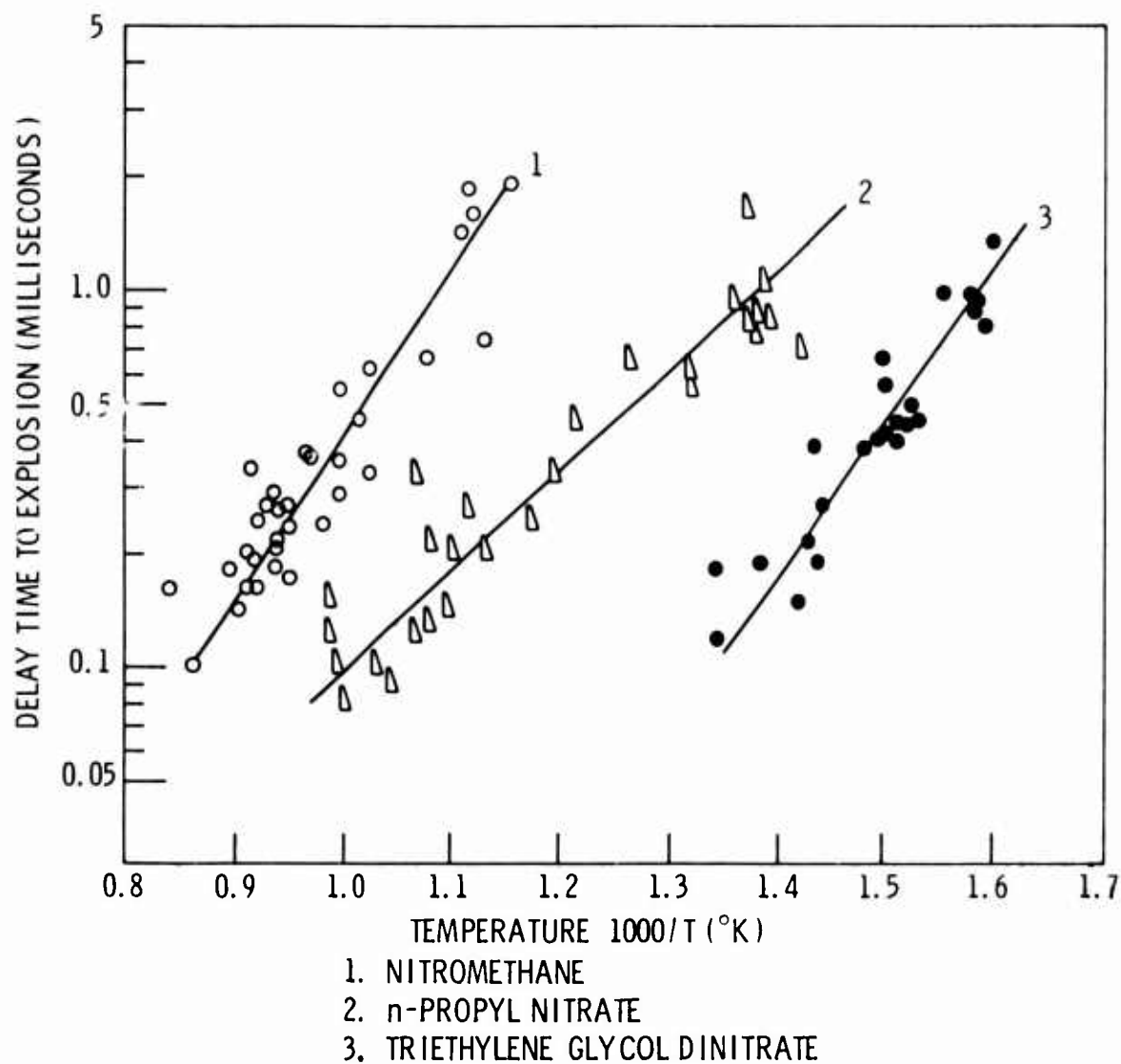


FIG. 5 THERMAL INITIATION DATA

TABLE I

THERMAL INITIATION TEMPERATURE
250 MICROSECOND HEATING TIME

<u>Compound</u>	<u>Temperature, °C</u>
Nitromethane	780
n-Propyl Nitrate	593
Triethylene Glycol Dinitrate	422

APPENDIX A

DETAILED CIRCUIT OPERATION

This detailed description is included as a construction or maintenance guide for the thermal initiation apparatus. Refer to the fold-out circuit diagram Figure A-6 and Table A-II for the following discussion.

The apparatus is ac powered by switches S1, S2, and a front panel switch on power supply PS3. There is a four minute delay for heating the thyatron (5C22) filament after which time the pilot light P2, indicates ac power is available for the high voltage power supply PS1. Before activating PS3 its current control is adjusted for about 1A so that the 16,000 μ F bank will not impose an excessive load on PS3. After charging the 16,000 μ F bank the current control is set at maximum level.

The charge push button switch S3 can now be depressed to bring the ac power to PS1, the "delay timer", the variac T2, and the self locking relay Re6. Relay Re6 immediately by-passes S3 thus effectively taking it out of the circuit. (Note switch S9 in the firing chamber must be closed for Re6 to operate.) The variac T1 is set for the desired high voltage ranging from 300 to 3000 volts, and the "delay relay" is set for 25 seconds changing time.

At the end of this changing period the "delay timer" switches state, turning off the high voltage power supply and turning on the exhaust fan in the "firing chamber". With the switch S4 in the "on" or "auto-fire" position the "delay timer" also automatically controls the voltage calibration, the powering of R₀ in the "firing chamber", and the firing of the thyatron. Otherwise with the S4 open, S5 can be used to manually activate these sub-units.

The switching of the "delay timer" or S5 controls the triggering of the SCR (2N 1881) and energizes relay Rel. The output from the SCR triggers the CRO horizontal sweep for a zero volts reference trace. Rel switches about 20 msec after the switching of the "delay timer". Rel controls the energizing of Re2, Re3, and Re4. Re2 switches first, applying a calibration voltage to the -B input of the CRO vertical amplifier. Re4 switches about 1 msec later connecting PS3 to R₀ via the two 25 Ω parallel isolating resistors in the "firing chamber". Re3 switches about 1 msec later connecting the 1 μ F capacitor that is charged to the voltage of PS2 to the pulse transformer T6 for firing the thyatron. Rel returns to its original state about 20 msec after initially switching, thus returning relays Re2, Re3, and Re4 to their original open state. Note this means R₀ is powered by PS3 about 20 msec. Concurrently with the firing of the thyatron (5C22) for discharging the high voltage energy storage capacitor a capacitor divider from the energy storage capacitor provides a low

voltage pulse for triggering the horizontal CRO sweep and the counter interval timer. The CRO and camera display and record the signal across R_0 . The crystal microphone located next to R_0 picks up the detonation bang thus stopping the interval timer.

When the "firing chamber" cover is raised, three microswitches S7, S8, and S9 are opened, and ac power is removed from 3 relays, Re5, 9, and 6 respectively. For safety Re5 shorts out the energy storage capacitor. To measure the voltage drop across R_0 and thus determine its resistance Re9 connects both a low constant current circuit (≈ 50 ma) to the sample explosive resistor R_0 , and a L&N potentiometer. The self locking relay Re6 is also reset, and thus ac power is removed from the "delay timer", the variac T1, and the exhaust fan. All relays have now been returned to their original state, and with the closing of the firing chamber the unit is ready for another test firing.

TABLE A-II
COMPONENT FUNCTION AND DESCRIPTION

<u>Relays</u>		
	<u>Function</u>	<u>Description</u>
Re1	Delay	New Products, Inc - reed relay #162 coil - 6V at 700 ohms
Re2	Auto-Cal	Stevens Arnold, Inc - millisec relay coil - 18V at 1400 ohms
Re3	Thyratron Fire	C. P. Clare & Co - mercury relay type HQP-1002 coil - 12V at 4500 ohms
Re4	R ₀ Power	Same as Re3
Re5	Safety (Capacitor Discharge)	Struthers Dunn Co - power relay type 8BXX - coil 115V AC
Re6	Latching Relay for Capacitor Charge	Advance Electric & Relay Co type A-2041419 coil 115V AC
Re7, 8	Preheat Thyratron Filament 4 Minutes	Amperite - 6V N/O 120 sec
Re9	Resistance Measurement	Same as Re6 and with hermetic sealing

<u>Switches</u>		
S1	AC Power	SPST
S2	AC Power	DPDT
S3	Capacitor Charge	Pushbutton N/O
S4	Automatic Fire	SPST
S5	Manual Fire	Pushbutton N/O
S6	Static Calibration	SPST

Switches (Continued)

	<u>Function</u>	<u>Description</u>
S7	De-energize Re5 to Discharge Capacitor When Firing Chamber Open or Power Failure	Microswitch SPST N/O
S8	De-energize Re7 for Resistance Measurement When Firing Chamber Open	Microswitch SPST N/O
S9	Reset Re6 When Firing Chamber is Opened	Microswitch N/O

Timer

Delay Timer	Control Capacitor Charging Time, etc	Syracuse Electronics Corp TR-302 DPDT-5A-N/O
-------------	--------------------------------------	---

Power Supplies

PS#1	Universal Voltronics Corp BPE-10-5.5 (0-10KV at 5.5 ma)	
PS#2	Sorensen Model QM/48V at 40 ma	
PS#3	NJE Corp Model SY-60-6-M (10-60V @ 6 amp)	

Transformers

T1	High Voltage Control	GR Variac at 1 amp
T2	Filament	Stancor P6308 - 6.3 V AC at 10 amp
T3	Thermal Delay Power	6.3 V AC at 1.2 amp
T4	Fine AC Control for Incremental Changes	2.5V AC at 3 amp
T5	of PS#1	GR Variac at 1 amp
T6	Trigger Thyatron	Pulse Engineering #2982

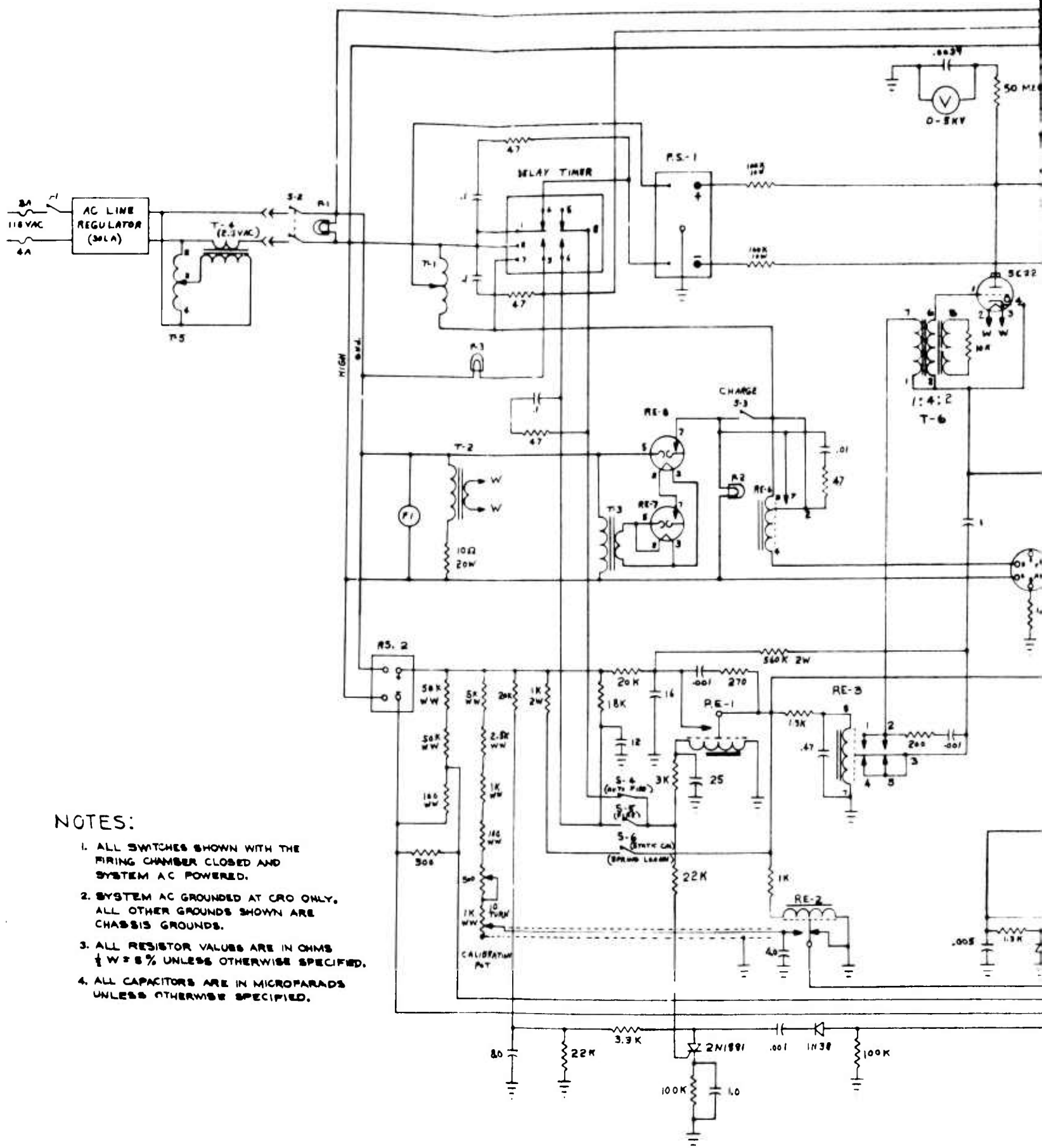
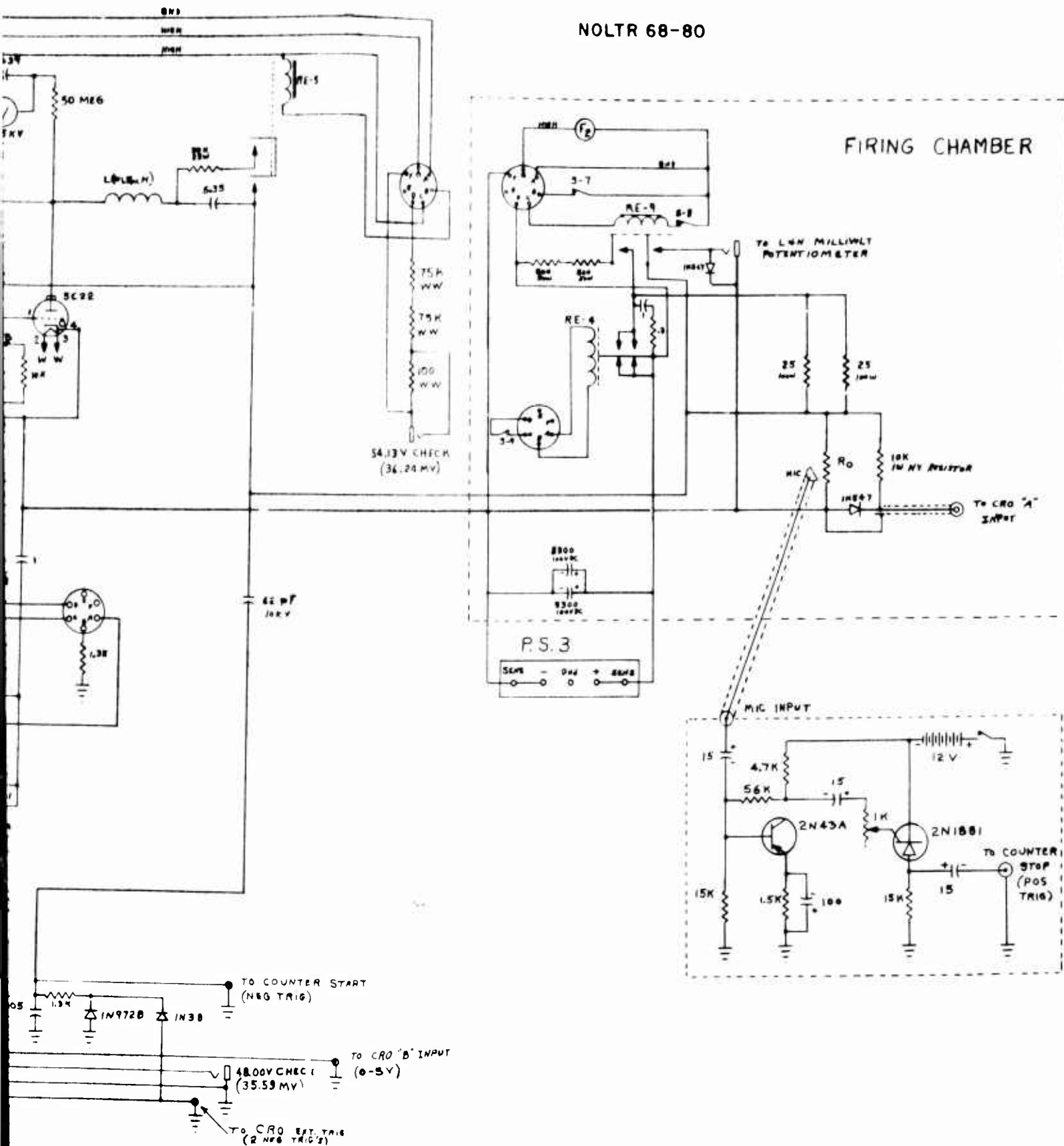


FIGURE A-6 THERMAL INITIATION APPARATUS



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Kendall and Jerome Rosen. 19 June 1968.
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A thermal initiation apparatus is fully
described for rapidly heating and monitoring
high energy materials in the range of 300
to 1000°C. Time delays to explosion, which
range from 0.05 to 5 msec are a function of
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